Patent
Docket No.: 158-P-C1553US

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Application of: Peter Kuhlmann and Reinhard Winter

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For: ALKYL RESIN EMULSIONS AND UTILIZATION OF THE SAME

Group Art Unit: 1714
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Examiner: Patrick Dennis Niland

**CERTIFICATE OF FACSIMILE TRANSMISSION UNDER 37 C.F.R. 1.8:** I hereby certify that this correspondence is being sent by facsimile to the telephone number shown below, addressed to the Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on the below indicated date:

Facsimile Number: 703-872-9310
Date: February 17, 2004 By: Janelle K. Grube

**DECLARATION UNDER 37 CFR §1.132 of Peter Kuhlmann**

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

I, Peter Kuhlmann, hereby declare that:

1. I am the same Peter Kuhlmann who is identified as a co-inventor in the above-identified application ("our application").

2. In 1977 I received a PhD degree in Chemistry from the Technische Universität Clausthal in Clausthal-Zellerfeld, Germany.

3. From 1978 to the present I have been employed by Ashland-Südchemie-Kernfest GmbH, where I have performed work involving paints and coatings. My current title is Business Manager.

4. I am a co-inventor of U. S. Patent No. 4,772,490.

5. I have read the Final Rejection mailed June 4, 2003 and the Office Action mailed November 18, 2003, cited U.S. Patent No. 3,639,315 (Rodriguez) and the German priority document for our application.

6. Rodriguez does not anticipate our claimed two-component paint system. Rodriguez describes a urethane-modified version of the air-drying or baking single-component paints discussed at page 2, lines 21 – 27 of our application. Rodriguez modifies a hydroxyl-containing resin by reaction with an isocyanate to form "urethane-modified, water-dispersible resins" (see for example col. 1, lines 65-67, col. 4, lines 69 – 71 and col. 5, lines 59 – 60). The hydroxyl-isocyanate reaction is carried out "until substantially all of the organic isocyanate has reacted" (see for example col. 2, lines 16 – 19). The resulting product is a single-component paint that forms a stable dispersion in water (see for example col. 1, lines 57 – 62) and which can be stored in liquid form for long periods of time if it is not exposed to significant amounts of oxygen. When Rodriguez's single-component paint is applied in a thin film to a substrate, curing takes place by reaction of ambient oxygen with available double bonds in the unsaturated fatty acid portion of the water-dispersible resin. This reaction of ambient oxygen with the available double bonds is what Rodriguez refers to when he says that his products are "capable of air drying in a short time", see col. 1, lines 18 – 20.

7. To further explain the differences between our claimed two-component paint system and Rodriguez, the following experiments were carried out by me or under my direction. The experiments compared our Example 4 formulation and Rodriguez's Example 2 and 6 formulations. Our Example 4 formulation is an unpigmented two-component paint, whereas Rodriguez's Example 2 and 6 formulations were unpigmented single-component paints. Rodriguez's Example 2 and 6 formulations respectively represent Rodriguez's lowest (7% based on alkyd solids) and highest (16% based on alkyd solids) exemplified toluene diisocyanate (TDI) addition levels. Each formulation was mixed thoroughly and allowed to stand for 10 minutes before application to a substrate, or mixed for 60 minutes (as was done in Rodriguez Example 2) and allowed to stand 24 hours before application. The coated substrates were dried in an oven for 1 hour at 80° C, tested 24 hours later to adjust the samples to the environmental conditions under which the samples finally were tested, and evaluated as shown below:

| Example | Adhesion 1 | 20° Gloss[2] | 60° Gloss[2] | Water Resistance | Acetone Resistance | Methyl Ethyl Ketone Resistance |
|---|---|---|---|---|---|---|
| Our Example 4, 10 minute standing time | TT 0 | 88 | 96 | > 240 hours | > 100 double rubs | > 100 double rubs |
| Our Example 4, 24 hour standing time | TT 5 | 1.5 | 12 | < 1 minute | 2 double rubs | 2 double rubs |
| Rodriguez's Example 2, 24 hour standing time | TT 1 | 88 | 94 | 60 minutes | 3 double rubs | 3 double rubs |
| Rodriguez's Example 6, 24 hour standing time | TT 5 | 58.2 | 91.8 | 60 minutes | 12 double rubs | 12 double rubs |

1 On a TT 0 to TT 5 scale in which TT 0 signifies no failure and TT 5 signifies total failure.

2 On a 0 to 100 scale in which higher values represent higher gloss.

The above table clearly shows that our example is advantageous compared to Rodriguez' examples and to our example performed according to the teaching of Rodriguez. The above results show that the two-component paint of our Example 4 is not the same as and has much better solvent resistance (not only water but also Acetone and Methyl Ethyl Ketone Resistance) than Rodriguez's Example 2 and Example 4 single-component paints. Furthermore, the adhesion and gloss properties are better.

Applicant believes that the difference in properties is caused i.a. by the fact that in Rodriguez the isocyanate has substantially reacted with the alkyd resin before the paint is applied to the substrate (see also claim 1 of Rodriguez section 3). The curing of the paint according to Rodriguez on the substrate is done by cross-linking of the unsaturated double bonds by means of air oxygen.

Contrary to Rodriguez the two-component composition according to the present application is applied to a substrate before a substantial part of the isocyanate has reacted with the alkyd resin. The curing according to the present application is done by the reaction of isocyanate with the alkyd resin.

If one applies the teaching of Rodriguez (substantially reacting the isocyanate with the alkyd resin before application to a substrate) to the present application (see our Example 4, 24 hours standing time), the properties of the paint are poor.

8. To further explain the differences between our claimed two-component paint system and Rodriguez, I note that our claim 1 recites (a) a "two-component paint system", (b) in which "the first component and second component are formulated to provide a paint containing at least a stoichiometric ratio of isocyanate groups to hydroxyl groups", and (c) "with no more than 30% of the hydroxyl groups being pre-reacted with isocyanates". I will refer to these quoted phrases as "Item (a)", "Item (b)" and "Item (c)", and compare them to Rodriguez's working examples.

9. Rodriguez's Example 1 describes an alkyd resin whose hydroxyl content can be calculated at 4.07 wt. %. The Rodriguez Example 1 resin is the starting alkyd resin for Rodriguez to be modified with isocyanate and, therefore, not a two-component paint system (items (a) – (c) are not fulfilled).

10. Rodriguez's Example 2 describes a single-component paint formed by reacting a water dispersion of the Rodriguez Example 1 resin with TDI. The TDI would have had 49 wt. % isocyanate content (if a Technical Grade) or 51.2 wt. % isocyanate content (if a Research Grade). Applying the basic isocyanate value formula from page 7, lines 29 – 31 of our application and a worst-case (Research Grade or highest isocyanate content TDI) assumption to Rodriguez Example 2, the basic isocyanate value would be:

$$\text{basic isocyanate value} = \frac{42 \times 100 \times 4.07 \text{ wt. \% hydroxyl in the second component}}{17 \times 51.2 \text{ wt. \% isocyanate in the first component.}}$$

$$= 19.64$$

Thus 19.64g of Research Grade TDI would be equivalent to 100g of the Rodriguez Example 1 alkyd resin solids. Rodriguez Example 2 added 7g (7%) TDI per 100g alkyd resin solids. This was less than the basic isocyanate value and less than a stoichiometric ratio of isocyanate groups to hydroxyl groups. The Rodriguez Example 2 paint is not a two-component paint system (see Item (a)) and is not a paint containing at least a stoichiometric ratio of isocyanate groups to hydroxyl groups (see Item (b)).

11. Rodriguez's Example 3 describes two pigmented paints, namely a paint formed by adding pigment to the Rodriguez Example 1 unmodified resin and a paint formed

by adding pigment to the Rodriguez Example 2 urethane-modified resin. As already explained in section 9 above, the first Rodriguez Example 3 paint is not a two-component paint system (since no isocyanate is added none of items (a) –(c) apply). The second Rodriguez Example 3 paint is not a two-component paint system either (see Item (a)) and is not a paint containing at least a stoichiometric ratio of isocyanate groups to hydroxyl groups (see Item (b)).

12. Rodriguez's Example 4 describes a single-component paint formed by reacting a water dispersion of the Rodriguez Example 1 resin with phenyl isocyanate. Applying the above-mentioned basic isocyanate value formula and assuming 35.3 wt. % isocyanate content for phenyl isocyanate yields a basic isocyanate value of 28.5. Thus 28.5g of phenyl isocyanate would be equivalent to 100g of the Rodriguez Example 1 alkyd resin solids. Rodriguez Example 4 employed 10g (10%) phenyl isocyanate per 100g alkyd resin solids. This was less than a stoichiometric ratio of isocyanate groups to hydroxyl groups. The Rodriguez Example 4 paint is not a two-component paint system (see Item (a)) and is not a paint containing at least a stoichiometric ratio of isocyanate groups to hydroxyl groups (see Item (b)).

13. Rodriguez's Example 5 describes a single-component paint formed by reacting a water dispersion of the Rodriguez Example 1 resin with 4,4'-methylene-bis(cyclohexyl isocyanate. Applying the above-mentioned basic isocyanate value formula and assuming 33.6 wt. % isocyanate content for 4,4'-methylene-bis(cyclohexyl isocyanate yields a basic isocyanate value of 29.9. Thus 29.9g of 4,4'-methylene-bis(cyclohexyl isocyanate would be equivalent to 100g of the Rodriguez Example 1 alkyd resin solids. Rodriguez Example 5 employed 7g (7%) 4,4'-methylene-bis(cyclohexyl isocyanate per 100g alkyd resin solids. This was less than a stoichiometric ratio of isocyanate groups to hydroxyl groups. The Rodriguez Example 5 paint is not a two-component paint system (see Item (a)) and is not a paint containing at least a stoichiometric ratio of isocyanate groups to hydroxyl groups (see Item (b)).

14. Rodriguez's Example 6 describes an alkyd resin whose hydroxyl content can be calculated at 2.85 wt. % and a single-component paint formed by reacting a neat solution of this alkyd resin with TDI. Applying the above-mentioned basic isocyanate value formula and worst-case (Research Grade TDI) highest isocyanate content assumption, the basic isocyanate value would be 13.75. Thus 13.75g of Research Grade TDI would be equivalent to 100g of the Rodriguez Example 6 alkyd resin solids. Rodriguez Example 6 employed 16g (16%) TDI per 100g alkyd resin solids, an amount that was slightly more than the basic isocyanate value. However, Rodriguez added the TDI in two stages, by (i) making a 9.8% TDI addition to the neat alkyd resin and allowing the mixture to react at 180° F (82° C) for one hour, followed by (ii)

dispersing the resulting product in water, adding enough additional TDI to give a 16% modification and allowing the mixture to react at 75° F (24° C) for 10 minutes. The first TDI addition was performed under conditions that would pre-react substantially all available hydroxyl groups with isocyanate. For example, the resin was not dispersed in water, thus preventing such water from competing with hydroxyl groups for reaction with the TDI. The resin had just been heated to 420° F (215° C) and then cooled to 150° F (66° C), which would remove substantially any other water that may have been present. The 180° F (82° C), one hour reaction conditions following the first TDI addition would ensure substantially complete reaction of the hydroxyl groups in the resin with isocyanate. The Rodriguez Example 6 paint is not a two-component paint system (see Item (a)) and is not a paint with no more than 30% of the hydroxyl groups being pre-reacted with isocyanates (see Item (c)).

15. To further explain the differences between our claimed two-component paint system and Rodriguez, I note that in our Example 1 we formed a 45 wt. % solids alkyd resin (see page 8, line 26) whose hydroxyl content can be calculated at 4.48 wt. %, neutralized the resin with triethylene amine and emulsified the neutralized resin in water. In Example 2 we mixed the Example 1 emulsion with pigments to form a white parent paint containing 60 Parts of the alkyd resin emulsion in 97 parts of the parent paint (see page 9, lines 1 – 5), or 28 wt. % alkyd resin. We then formed two two-component paints as follows:

16. In our Example 3 we mixed the Example 2 parent paint at a 4:1 mixing ratio with a curing agent containing 70 wt. % of a polyisocyanate based on hexamethylene diisocyanate (see page 9, lines 11 – 16) and having a 22 wt. % isocyanate content, to form a paint/curing agent mixture having a processing time of about 4 hours (see page 9, line 17). Thus in our Example 3 we mixed 100 parts of the parent paint (28 parts of which were the alkyd resin solids) with 25 parts of the curing agent (70 wt. % or 17.5 parts of which were the polyisocyanate). Applying the above-mentioned basic isocyanate value formula yields a 50.3 basic isocyanate value for the components in our Example 3. Thus 50.3g of the Example 3 polyisocyanate would be equivalent to 100g of the Example 1 alkyd resin solids. We combined 17.5 parts of the polyisocyanate with 28 parts of the alkyd resin solids, or 64.8g isocyanate for 100g alkyd resin solids. This was a 30 percent isocyanate excess (64.8/50.3) over the basic isocyanate value. Our Example 3 paint/curing agent mixture was a two-component paint system (see Item (a)), containing at least a stoichiometric ratio of isocyanate groups to hydroxyl groups (see Item (b)) and which did not have more than 30% of the hydroxyl groups pre-reacted with isocyanates (see Item (c)).

17. In our Example 4 we formed an unpigmented mixture containing 87 wt. % of the Example 1 emulsion, or 39.2 wt. % alkyd resin solids. We combined this unpigmented mixture at a 3:1 mixing ratio with the Example 2 curing agent. Thus we mixed 100 parts of the unpigmented mixture (39.2 parts of which were the alkyd resin solids) with 33.3 parts of the curing agent (70 wt. % or 23.3 parts of which was the polyisocyanate). This represented 59.4g isocyanate for 100g alkyd resin solids, and was an 18 percent isocyanate excess (59.4/50.3) over the basic isocyanate value. Our Example 4 mixture was a two-component paint system (see Item (a)), containing at least a stoichiometric ratio of isocyanate groups to hydroxyl groups (see Item (b)) and which did not have more than 30% of the hydroxyl groups pre-reacted with isocyanates (see Item (c)).

18. All statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the Application or any patent issuing thereon.

Further Declarant saith not.

Dec. 4th, 2003
Date

Peter Kuhlmann